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A Small, Inexpensive Apparatus for the Determination
of the Density of Powdered Materials

by

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"A Small, Inexpensive Apparatus for the Determination of the
Density of Powdered Materials"

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ABSTRACT

A small inexpensive apparatus for measuring the density of powders at an accuracy of 1% or better is presented. Since the working medium is a gas, the density of air or solvent sensitive samples can easily be measured with this apparatus in a glove box.

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The measurement of density by the conventional liquid immersion technique (1) for materials that are powders is usually difficult to perform and prone to large errors. The main problems are caused by gas trapped between particle grains, the lack of wetting of the solid by the liquid, and possibly chemical reaction between the solid and the liquid. Many of these problems can be overcome by using a gas as the working medium; however, existing gas methods are time consuming (2). Here we present a simple apparatus to rapidly determine the density of powders with moderate accuracy.

Knowledge of the density of a new material is often useful information. For example, the density is an important parameter for crystal structure determination. After the unit cell dimensions are obtained by x-ray diffraction and the empirical formula is determined by chemical analysis, the number of chemical formula units per unit cell can be determined from the density. If the material of interest is a solid state compound composed of elements of quite different atomic weights AND if the unit cell is small, the contents of the unit cell can sometimes be determined from the measured density; that is, the empirical formula may be obtained without a direct chemical analysis. Since the number of formula units contained in a unit cell is very often a small integer, the density does not have to be determined with high accuracy; usually 1 or 2 percent is more than sufficient.

While methods to determine the density of very small amounts of material (0.1 mg) have been developed (1), these are difficult to perform, especially on air sensitive samples. For example, the single crystals used in x-ray crystallography are quite small (sometimes less than 0.1mm in any dimension). However, the density could be determined using our apparatus if a large number of crystals were available, as is often the case. New materials are often first prepared in polycrystalline powder form and the subsequent preparation of a nonporous solid mass or single

crystal may be very difficult or even impossible, so that the density must be determined from such powders.

There are several different existing methods for density determination. The oldest (Archimedes method) is to weigh a material and measure its volume by determining the volume of liquid it displaces. Variations on the method include pycnometric or hydrostatic techniques (1). Finally, a method for powders based upon the compression of a gas in a standard volume containing the sample has also been used (2). In all the above methods there is one underlying assumption: the material does not include voids (except perhaps for atomic vacancies). In such a case, all the above methods will result in density values that are less than the true density of the solid itself. Of course, in materials of known density the measurement would directly give the void volume. Here we report on a variation of the powder method that easily determines density of small samples to about 1%. The principle of the method is outlined next.

A standard closed volume is connected to a pressure transducer and to a calibrated piston. The closed volume is formed by covering a hole drilled in a metal block with a plate and o-ring so that a sample may be included inside (see figure 1). The apparatus is first calibrated when the closed volume contains only gas (near atmospheric pressure) and no sample. The piston is moved a fixed distance to produce a volume change.

$$dV = V_1^e - V_2^e \quad (\text{eq. 1}),$$

where V_1^e is the initial volume of the empty container and V_2^e is the final volume. As long as the volume change is not too large, so that the pressure remains at or below 1 or 2 atmospheres, the ideal gas law may be used to relate the pressure and the volume. We assume the use of an appropriate gas so that this is true. At room temperature gasses such as O_2 , N_2 , He, and Ar obey the ideal gas law in this pressure range to about 0.1% (3). In that case we

In that case we have:

$$P_i V_i^e = nRT = P_f V_f^e \quad (\text{eq. 2}),$$

where P_i and P_f are the initial and final pressure read from the transducer. Combining eq. 1 and 2 allows the determination of the initial volume and the final volume:

$$V_i^e = dV / (1 - P_i/P_f) \quad (\text{eq. 3})$$

and then V_f^e is obtained from eq. 1. The volume of the system is now calibrated. This method assumes a method or apparatus that allows an accurate control and measurement of the volume change, dV , caused by the piston movement. A method of determining the system volume without having a direct measure of dV but with good control of the piston position will be discussed a little later in the article.

Now that the system is calibrated, a powdered sample is introduced into the sample chamber that is a part of the system volume (see figure 1). The pressure is now measured with the piston in the same initial and final positions as used above to calibrate the volume. Thus the initial volume of the cell including the sample is:

$$V_i^s = dV / (1 - P_i^s/P_f^s) \quad (\text{eq. 4})$$

where the superscript s indicates the volume and pressures of the gas when the sample is included. The density is obtained by dividing the weight of the sample by the volume of the sample, $V = V_i^e - V_i^s$ (or by a similar equation involving the final volumes).

If the volume change dV is not easily measurable, but the piston position can be accurately controlled, then the initial empty volume of the system can be determined by using a sample of known density as a standard. Both equation 1 and 2 are used with dV and V_i^e as unknowns, and a known sample volume, $V = V_i^e - V_i^s$,

equal to the weight of the sample divided by the known density. With two separate measurements, one with an empty cell and a second with the standard material in the cell, the two unknowns can be determined.

The key parts of the apparatus are the piston and the pressure transducer. For the former we used a calibrated 2.5 milliliter glass syringe with a teflon tipped piston (manufactured by Hamilton , model number 1002-LT). The connector at the end for attaching a needle was cut off. The teflon piston seals very effectively to the glass body of the syringe so that a pressure of 2 atmospheres is held with no observable change for over one hour. A small ammount of vacuum grease was applied to the teflon in order to keep a low sliding friction.

The transducer is an inexpensive (approx. \$50) silicon strain gauge device (Omega Engineering, model PX 136-015G V). The transducer has an output that is close to linear in pressure. Approximately 120mV output is obtained at one atmosphere gas pressure when the transducer is powered by a 12V battery source; approximately 0mV output is obtained at zero pressure. Both the transducer and the syringe body are epoxied into a 2.0" diameter brass block that has been drilled out as shown in figure 1. The base plate of the apparatus has a sample container and an o-ring to form a reproducible seal to the brass block. The base plate is fastened to the block by four 6-32 Allen head screws. A small modification of the design to make a side loading holder for the sample would result in somewhat easier handling during loading of the cell.

The piston is moved by turning a well machined screw or micrometer head. Readings are made only when turning the screw in one direction, to avoid most of the mechanical hysteresis in the system. In our case we used a 0.25" diameter stainless steel rod that was machined to a 40 turn per inch screw (available from Tropel, Inc under the name of Unislide, but could easily be

manufactured in most machine shops). Alternatively, a micrometer head could be used for the same purpose.

The main sources of experimental error are the zero offset of the transducer (approx. $\pm 2\text{mV}$), the temperature dependence of its output at constant pressure (approx. $1\text{mV}/^\circ\text{C}$ at one atmosphere pressure), and small non-linearities in the transducer response. It is relatively easy to keep the temperature of the apparatus constant to about 0.1°C during the measurement time of approximately 5 or 10 minutes. The zero offset and non-linearity can be measured directly if the apparatus is connected to a calibrated variable pressure source or "ignored" so that it is compensated for in the calibration using a standard material of known density.

The initial calibration of the empty closed volume was performed using about 0.2cc of -100 mesh powdered silicon. With the dimensions shown in figure 1 and a piston compression stroke of approximately $0.625"$, we obtained a dV of 0.662cc and an empty volume, V_1^e of 1.360cc (piston in the expanded position). The value of dV agreed with the estimate obtained by reading the milliliter markings on the syringe, but could be calculated to a higher precision than could be estimated from reading the markings. Repeated measurements of these two volumes suggested that they could be determined to 0.2% . In this calibration we assumed exact linearity of the transducer output voltage with pressure and a transducer offset voltage of 0.0mV . The pressure change was less than a factor of two, with the initial pressure (piston in expanded position) close to one atmosphere. In order to check the overall system accuracy using the above assumptions, we also measured the density of about 0.20cc of Cu powder (-400 mesh) and Pb shot (about 2mm diameter). The following densities (true values in parenthesis) were obtained: Cu 8.92g/cc (8.96) and Pb 11.36g/cc (11.34).

This demonstrates that with very little precaution and the

simplest assumptions concerning the transducer characteristics, the density of powders can be determined to better than 1% accuracy. Although we have not pushed the technique further to improve the overall accuracy, it seems quite likely that an accuracy of 0.2% could easily be attained by measuring the exact transducer characteristics and by carefully controlling the system temperature. We have used this apparatus to measure the density of powders both in the laboratory atmosphere and in an argon filled glove box and have found it to be robust and easy to use. A typical measurement of density, including the calibration of the empty volume, takes less than 15 minutes.

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We thank A. H. Thompson for initially suggesting a gas technique for measuring the density of powders, a suggestion that eventually lead to the present design. We also thank David Peale for some helpful suggestions. Finally we thank those who supported this work, the Office of Naval Research and the Office of Basic Energy Sciences of the Department of Energy.

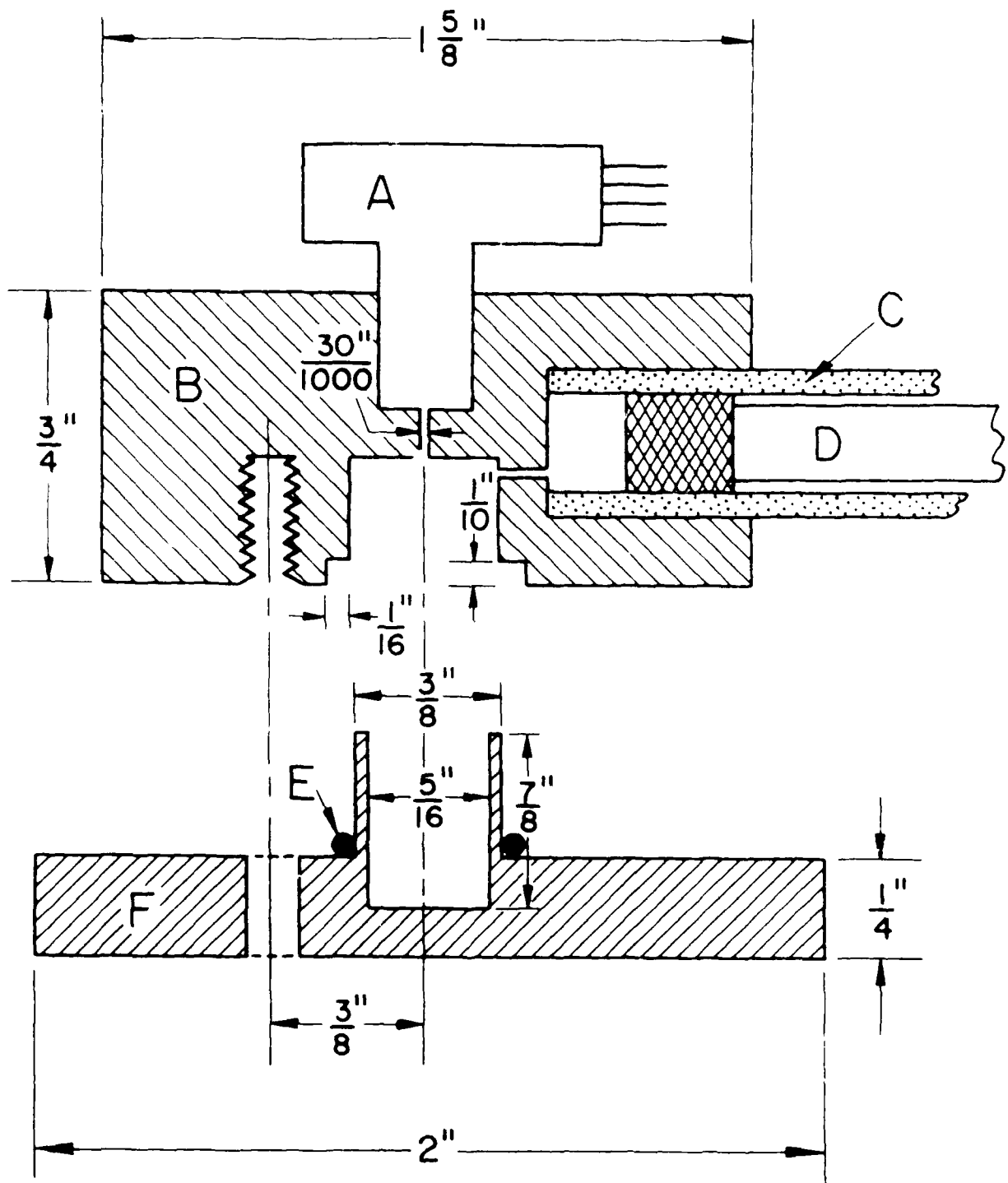
FIGURE CAPTIONS

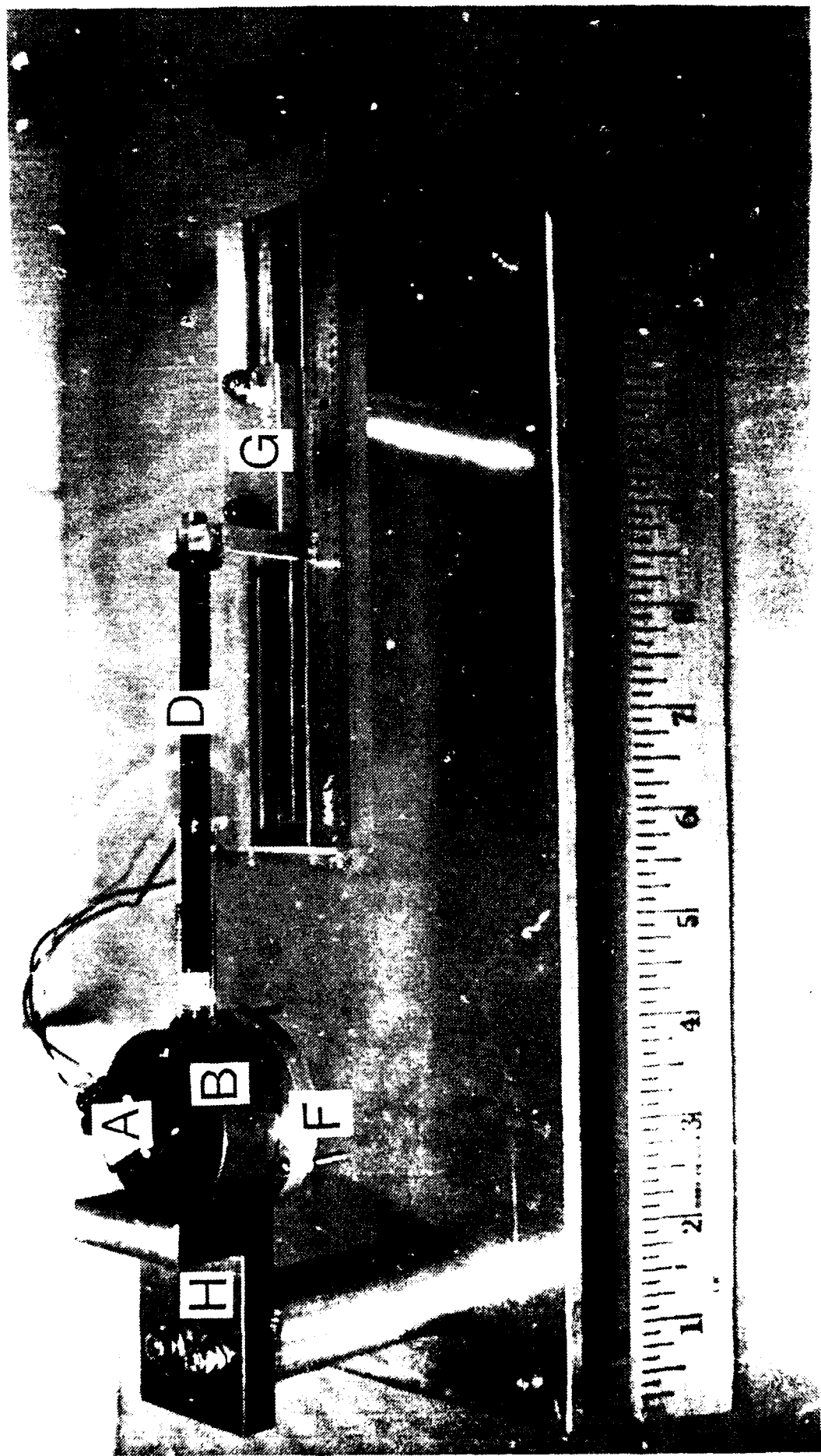
1. A schematic diagram of the density apparatus shows the important working parts: A - strain gauge pressure transducer, B - brass body of the top section of the cell, C - glass body of the syringe, D - syringe plunger, E - #12 o-ring to seal the upper and lower cell sections when assembled, F - aluminum base of the cell. The strain gauge and the syringe body are epoxied to the cell. The base is fastened to the top section by four 6-32 Allen head screws. The sample fills the cup in the lower aluminum section of the cell.

2. A photograph of the entire apparatus shows the size and placement of the parts. The large ruler is marked in inches. The parts are labeled as in figure 1: A - pressure transducer, B - top section of the cell, D - syringe plunger, F - cell base, G - sliding fixture to push plunger (the plunger moves 0.025" per turn of the machine screw by the nob at the far right), H - angle support for the top section of the cell.

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